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IS 1767 (1980): Dicalcium Phosphate for Dentifrice [PCD 19: Cosmetics]

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Indian Standard

SPECIFICATION FOR DICALCIUM PHOSPHATE FOR DENTIFRICE

(Second Revision)

First Reprint AUGUST 2007
(Including Amendment No. 1 & 2)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 5 *February* 1981

AMENDMENT NO. 2 JULY 2004
TO
IS 1767 : 1980 SPECIFICATION FOR DICALCIUM PHOSPHATE FOR DENTIFRICE

(Second Revision)

[*Page* 5, *Table* 1, *Sl No.* (iii), *col* 2] — Substitute the following for the existing:

'Dicalcium phosphate (as $CaHPO_4$), percent by mass, *Min* (on dry basis) 98.0'

(*Page* 5, *Table* 1) — Insert the following at the end:

(1)	(2)	(3)	(4)
'xvi)	Proteinous impurities	To pass test	A-17
xvii)	Reducing substances	To pass test	A-18'

(*Page* 15, *clause* **A-16.3**) — Include the following at the end:

A-17 TEST FOR PROTEINOUS IMPURITIES

A-17.1 Procedure

Heat 0.5 g of the material gently in a dry test tube. The material shall be taken to have passed the test, if no change in colour and no emission of any unpleasant odour is observed.

A-18 TEST FOR REDUCING SUBSTANCES

A-18.0 Outline of the Method

The colour produced with potassium permanganate is matched against that obtained with standard calcium carbonate solution.

A-18.1 Apparatus

A-18.1.1 *Nessler Cylinders* — 50-ml capacity.

A-18.2 Reagents

A-18.2.1 *Concentrated Sulphuric Acid*, 3 M.

A-18.2.2 *Potassium Permanganate*, 0.005 M.

A-18.2.3 *Calcium Carbonate* (*Analytical Grade*)

A-18.3 Procedure

Shake 1 g of the material with 5 ml of distilled water and 5 ml of 3 M Sulphuric acid for one minute, again add 20 ml of distilled water. Add 0.1 ml of 0.005 M potassium permanganate and shake for 15 s. Carry out a control test using 1 g of calcium carbonate in place of material and the same quantities of other reagents. Compare the colour produced in the two tubes.

A-18.3.1 The material shall be taken to have passed the test if the intensity of colour produced in the test with the material is not greater than that produced in the control test.

(PCD 19)

AMENDMENT NO. 1 FEBRUARY 1997
TO
IS 1767 : 1980 SPECIFICATION FOR DICALCIUM PHOSPHATE FOR DENTIFRICE

(Second Revision)

(*Page* 3, *clause* 0.4) — Substitute 'IS 460 (Part 1) : 1985*' *for* 'IS : 460 (Part I) - 1978*', 'IS 460 (Part 2) : 1985†' *for* 'IS : 460 (Part II) - 1978†' and 'IS 460 (Part 3) : 1985‡' *for* 'IS : 460(Part III) - 1978‡'.

(*Page* 3, *foot-notes with* '*', '†' *and* '‡' *marks*) — Substitute the following for the existing matter:

'*Test sieves: Part 1 Wire cloth test sieves (*third revision*).

† Test sieves : Part 2 Perforated plate test sieves (*third revision*).

‡Test sieves: Part 3 Methods of examination of apertures of test sieves (*third revision*).'

[*Page* 5, *Table* 1, *Sl No.*(xi)] — Substitute '400' *for* '50'.

(*Page* 6, *clause* A-1.1) — Substitute 'IS 1070 : 1992*' *for* 'IS 1070 : 1977*'.

(*Page* 6, *foot-note with* '*' *mark*) — Substitute the following for the existing matter:

'*Reagent grade water (*third revision*).'

(*Page* 7, *clause* A-4) — Substitute the following for the existing matter:

'A-4 DETERMINATION OF DICALCIUM PHOSPHATE

A-4.0 Outline of the Method — The material is dissolved in water using hydrochloric acid, which is then treated with disodium ethylenediamine-tetra-acetate and titrated with sodium hydroxide solution using hydroxynaphthol blue as indicator.

A-4.1 Reagents

A-4.1.1 *Hydrochloric Acid*

A-4.1.2 *Hydroxynaphthol Blue — Indicator*

A-4.1.3 *Triethanolamine*

(*Continued from page* 1)

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Indian Standard

SPECIFICATION FOR DICALCIUM PHOSPHATE FOR DENTIFRICE

(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 6 October 1980, after the draft finalized by the Cosmetics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was originally issued in 1961. Initially while reviewing this standard the Sectional Committee had agreed to indicate separately the essential and optional requirements. Subsequently, this was found non-implementable for certification and, therefore, a single set of requirements only was decided to be specified. All other changes considered necessary to align the standard with others in the series on raw materials for cosmetics have been introduced.

0.3 Dicalcium phosphate of interest to tooth paste manufacturers is almost invariably of the dihydrate type which has been suitably stabilized to ensure correct water of crystallization. However, another variation of dicalcium phosphate which is sometimes used in tooth paste manufacture is the anhydrous variety. Although this product is not included in the accepted dental remedies, it is nevertheless used because of its considerably higher abrasive power compared to the dihydrate.

0.4 For determination of fineness, IS sieves have been specified [*see* IS : 460 (Part I)-1978* IS:460 (Part II)-1978† and IS:460 (Part III)-1978‡].

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960§. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Specification for test sieves : Part I Wire cloth test sieve (*second revision*).

†Specification for test sieves : Part II Preforated plate test sieves (*second revision*).

‡Specification for test sieves : Part III Methods of examination of test sieves (*second revision*).

§Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for dicalcium phosphate for dentifrice.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of white powder without any taste or odour.

2.1.1 The material shall be almost insoluble in water but shall dissolve readily in dilute hydrochloric acid or dilute nitric acid. It shall be almost insoluble in alcohol.

2.2 The material when tested according to the methods prescribed in Appendix A, shall comply with the requirements given in Table 1. Reference to the relevant clauses of Appendix A is given in col 4 of the table.

NOTE — The requirements given in Table 1 are on the basis that no stabilizers have been added. When stabilizers have been added, the dicalcium phosphate content and some of the other characteristics are likely to get modified depending on the quantity and composition of the stabilizers.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in HDPE woven bags or as agreed to between the purchaser and the supplier.

3.2 Marking — The packages shall be securely closed and marked with the following information:

a) Manufacturer's name and/or his recognized trade-mark, if any;
b) Mass of the material;
c) Information whether it is stabilized or unstabilized;
d) Batch number, in code or otherwise to enable the date of manufacture to be traced back from records; and
e) Year of manufacture.

3.2.1 The product may also be marked with Standard Mark.

3.2.2 The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufactures or producers may be obtained from the Bureau of Indian Standards.

TABLE 1 REQUIREMENTS FOR DICALCIUM PHOSPHATE FOR DENTIFRICE

(*Clause* 2.2)

Sl No.	Characteristics	Requirement	Method of Test (Ref to Cl No. of Appendix A)
(1)	(2)	(3)	(4)
i)	Fineness:		
	a) Material passing through 75-micron IS Sieve, percent by mass, *Min*	99·9	A-2
	b) Material passing through 45-micron IS Sieve, percent by mass, *Min*	99·5	
ii)	Loss on ignition, percent by mass	24·0 to 27·0	A-3
iii)	Dicalcium phosphate (as $CaHPO_4.2H_2O$), percent by mass, *Min*	98·0	A-4
iv)	*p*H value	6·5 to 7·8	A-5
v)	Arsenic (as As_2O_3), parts per million, *Max*	2	A-6
vi)	Heavy metals (as Pb), parts per million, *Max*	20	A-7
vii)	Acid insoluble matter, percent by mass, *Max*	0·1	A-8
viii)	Bulk density, g/ml	0·75 to 1·00	A-9
ix)	Flow point*, ml	10 to 12	A-10
x)	Carbonates	To pass test	A-11
xi)	Iron (as Fe), parts per million, *Max*	50	A-12
xii)	Chlorides	To pass test	A-13
xiii)	Sulphates	To pass test	A-14
xiv)	Barium	To pass test	A-15
xv)	Fluorides (as F), percent by mass, *Max*	0·01	A-16

*Shall apply only for the material used for manufacture of tooth paste.

4. SAMPLING

4.1 The method for preparing representative test samples of the material and criteria for judging the conformity of the material to the requirements of this standard shall be as given in Appendix B.

APPENDIX A

(*Clause* 2.2)

METHODS OF TEST FOR DICALCIUM PHOSPHATE FOR DENTIFRICE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results.

A-2. DETERMINATION OF FINENESS

A-2.1 Apparatus

A-2.1.1 *Sieves* — 75-micron IS Sieve and 45-micron IS Sieve.

A-2.2 Procedure

A-2.2.1 Weigh accurately about 30 g of the material and transfer it to a 45-micron IS Sieve. Wash the material through the sieve by directing a slow stream of tap water upon it (*see* Note 1). Tapping may be necessary to start the water through the sieve. Continue washing the material until the washings are free from the portion of the material that is fine enough to pass through the sieve (*see* Note 2). Dry the sieve containing the residue on a steam-bath and finally in an oven at 105°±2°C. When dry, carefully brush the residue on to a tared watch-glass and weigh accurately.

NOTE 1 — Tap water is usually sufficiently free from suspended particles to be utilized directly as it comes from the faucet. If the water is turbid it is necessary to place a large bottle of distilled water above the working sink and to siphon the water through a clean rubber tube to the level of the sieve. It is a good plan to insert a small rubber stopper carrying a short glass tube about 5 cm in length and 0·6 cm in internal diameter in the faucet outlet. A thin flexible piece of clean rubber tubing (new tubing should first be thoroughly washed) about 60 cm in length is then attached to the glass tube. The faucet is turned on very slowly until a thin stream of water is coming through the rubber tube. By pressing the end of the rubber tube between the fingers, the stream of water may be varied in intensity and directed to any section of the sieve. Experience has shown that the best practice is to tilt and tip the sieve from one side to another alternately, and wash the sample from the higher level of the sieve to the lower.

NOTE 2 — In order to obtain comparable results, it is essential to continue washing the residue on the sieve until one is satisfied that all the material fine enough to pass through the sieve has been removed. At least half an hour of washing is essential in most cases. It is a good plan to catch the last washes in a beaker. By viewing the contents of the beaker against a dark coloured background, it is possible to gauge the progress of the washing operations accurately.

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*Specification for water for general laboratory use (*second revision*).

A-2.2.2 Transfer the residue in the watch-glass obtained in **A-2.2.1** to a 75-micron IS Sieve. Sieve it by washing with a stream of water as in **A-2.2.1** and then dry the final residue in the sieve on a stream-bath and then in an oven at 105° ± 2°C. When dry, carefully brush the residue on to a tared watch-glass and weigh again accurately.

A-2.3 Calculation

a) Residue on 45-micron IS Sieve, percent by mass $= 100 \frac{M_1}{M_3}$

b) Residue on 75-micron IS Sieve, percent by mass $= 100 \frac{M_2}{M_3}$

where

M_1 = mass in g of the residue in **A-2.2.1**,
M_3 = mass in g of the material taken for the test in **A-2.2.1**, and
M_2 = mass in g of the residue in **A-2.2.2**.

A-3. DETERMINATION OF LOSS ON IGNITION

A-3.1 Procedure — Weigh accurately about 1 g of the material and ignite at 850°C to constant mass. Calculate the loss in mass as percentage of the mass of the material taken for the test.

A-4. DETERMINATION OF DICALCIUM PHOSPHATE

A-4.0 Outline of the Method — The material is dissolved in a known quantity of hydrochloric acid, and the excess acid is estimated by titration.

A-4.1 Reagents

A-4.1.1 *Standard Hydrochloric Acid* — exactly 0·2 N.

A-4.1.2 *Standard Sodium Hydroxide Solution* — exactly 0·2 N.

A-4.1.3 *Methyl Orange-Sodium Indigo Disulphonate Indicator Solution* — Dissolve 0·1 g of methyl orange in hot water, filter and dilute to 100 ml with cold water. Separately dissolve 0·25 g of sodium indigo disulphonate in 100 ml of water and filter, if necessary. Mix equal volumes of the two solutions.

A-4.2 Procedure — Dissolve 1·000 g of the material in 50 ml of standard hydrochloric acid. Titrate the solution with standard sodium hydroxide solution, using 10 drops of the indicator to a greyish green end point.

A-4.3 Calculation

Dicalcium phosphate ($CaHPO_4.2H_2O$), percent by mass $= 3{\cdot}44 \, (50 - V)$

where

V = volume of standard sodium hydroxide solution required for titration.

A-5. DETERMINATION OF *p*H VALUE

A-5.1 Method — The determination shall be carried out either by the electrometric method or the indicator method. In case of dispute the electrometric method shall be adopted.

A-5.1.1 *Test Temperature* — The determination shall be made at room temperature and the material shall be tested in an atmosphere reasonably free from carbon dioxide.

A-5.2 Prepared Solution — Suspend 20·0 g of the material in 100 ml of freshly boiled and cooled distilled water. Shake the mixture for about 10 minutes. Allow to settle and use the supernatant liquid for the determination of *p*H value by the electrometric method or by the indicator method.

A-5.3 Electrometric Method — The determination shall be made with a *p*H meter.

A-5.4 Indicator Method

A-5.4.1 *Reagents*

A-5.4.1.1 *Bromocresol purple* — (*p*H range 5·2 to 6·8 and colour change yellow to bluish violet).

A-5.4.1.2 *Bromothymol blue* — (*p*H range 6·0 to 7·6 and colour change yellow to blue).

A-5.4.1.3 *Phenol red* — (*p*H range 6·2 to 8·4 and colour change yellow to red).

A-5.4.1.4 *Cresol red* — (*p*H range 7·2 to 8·8 and colour change yellow to red).

A-5.4.2 *Procedure* — Transfer 10 ml of the prepared solution (**A-5.2**) into each of four hard glass test-tubes and add 0·5 ml of the indicator, one to each tube. Compare the colour produced with a series of buffer tubes of known *p*H in the range 6·0 to 8·0. Report as *p*H, the *p*H of the buffer solution which gives the closest match with the colour produced with the prepared solution.

A-5.5 Standard calibrated glass discs may also be used for determination of *p*H.

A-6. TEST FOR ARSENIC

A-6.0 Outline of the Method — The stain on mercuric bromide paper produced by arsine is matched with that obtained with standard arsenic solution.

A-6.1 Reagent

A-6.1.1 *Dilute Hydrochloric Acid* — approximately 5 N.

A-6.2 Procedure — Dissolve 2·000 g of the material in the smallest possible amount of dilute hydrochloric acid until no more dissolves. Add water to make up to 10 ml. Transfer quantitatively to the Gutzeit bottle and carry out the test for arsenic as directed in IS : 2088-1971*, using 4 ml of standard arsenic trioxide solution for the composition stain.

A-7. TEST FOR HEAVY METALS

A-7.0 Outline of the Method — The colour produced with hydrogen sulphide solution is matched against that obtained with standard lead solution.

A-7.1 Apparatus

A-7.1.1 *Nessler Cylinders* — 50 ml capacity.

A-7.2 Reagents

A-7.2.1 *Dilute Hydrochloric Acid* — approximately 5 N.

A-7.2.2 *Dilute Acetic Acid* — approximately 1 N.

A-7.2.3 *Dilute Ammonium Hydroxide* — approximately 5 N.

A-7.2.4 *Hydrogen Sulphide Solution* — saturated.

A-7.2.5 *Standard Lead Solution* — Dissolve 1·600 g of lead nitrate in water and make up the solution to 1 000 ml. Pipette out 10 ml of the solution and dilute again to 1 000 ml with water. One millilitre of this solution contains 0·01 mg of lead (as Pb).

A-7.3 Procedure — Weigh 1·000 g of the material into a beaker. Add 3 ml of dilute hydrochloric acid and warm until no more dissolves. Dilute with water and make up the volume to 50 ml. Filter the solution. Transfer 25 ml of the filtrate into a Nessler cylinder. In the second Nessler cylinder, add 2 ml of dilute acetic acid, 1·0 ml of standard lead solution and make up the volume to 25 ml. Add 10 ml of hydrogen sulphide

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*Methods for determination of arsenic (*first revision*).

solution to each Nessler cylinder and make up the volume with water to 50 ml. Mix, allow to stand for 10 minutes and then compare the colour produced in the two Nessler cylinders.

A-7.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the second Nessler cylinder.

A-8. DETERMINATION OF ACID INSOLUBLE MATTER

A-8.1 Reagent

A-8.1.1 *Dilute Hydrochloric Acid* — approximately 4 N.

A-8.2 Procedure — Weigh accurately about 10 g of material in a beaker. Add about 50 ml of dilute hydrochloric acid. Heat for about half an hour on a water-bath. Then filter through an ashless filter paper, washing the residue with water until the washings are free of chloride ions. Dry the residue on the filter and weigh it.

A-8.3 Calculation

$$\text{Acid insoluble matter, percent by mass} = \frac{100A}{B}$$

where

A = mass in g of the residue, and

B = mass in g of the material taken for the test.

A-9. DETERMINATION OF BULK DENSITY

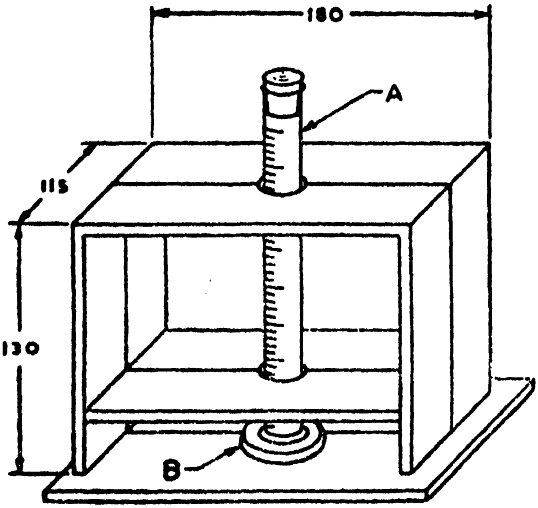
A-9.1 Apparatus

A-9.1.1 The apparatus is shown in Fig. 1. The base of the measuring cylinder A shall be ground flat and the empty measuring cylinder A together with the rubber bung shall weigh 250 ± 5 g. It shall be accurately calibrated to 250 ml with an error of less than 1 ml. The distance between zero and 250 ml graduation on the measuring cylinder shall be not less than 220 mm and not more than 240 mm. The distance between the flat ground part of the base of the measuring cylinder A and the rubber base pad B, when the measuring cylinder A is raised to the full height, shall be 25 ± 2 mm.

A-9.1.2 Balance — of a type providing easy access to the pans, the pans being at least 10 cm in diameter. It is necessary that the pointer should show a significant deflection for change in load of 0·25 g.

A-9.1.3 The rubber base pad B shall have a shore hardness of 35 to 50.

A-9.1.4 A timing device to indicate seconds.



All dimensions in millimetres.

Fig. 1 Apparatus for the Determination of Bulk Density

A-9.2 Procedure — Weigh about 42 g of the material on a piece of paper. Fold a piece of black glazed paper (25 × 25 cm) with two parallel creases to form a channel 12·5 mm down the middle of the paper. Counterpoise the paper on the balance and then place it flat on the bench. Place 75-micron IS Sieve on the black glazed paper in such a way that there is 50 mm space between the sieve and the paper. Transfer the weighed material to the sieve and with the back of the finger tips fitted with rubber finger-stalls, lightly rub all the powder through the sieve using a short stroke. Transfer the black glazed paper with the sieved material to the balance and adjust the mass of the sieved material to 40 g. Pick up the paper and form it into a chute. Allow it to lie between the thumb and fingers on the palm of the hand and introduce the material into the cylinder held at 45° to the vertical. Slip the powder gently and smoothly into the cylinder without knocking or squeezing. Assemble the measuring cylinder as shown in Fig. 1. With one hand gently grasp the upper part of the cylinder and within one second, lift it as far as 25 mm, do not jerk the cylinder by knocking it against the upper stop. At the start of the next second, release the cylinder smoothly. Continue lifting and dropping until 50 complete drops have been given. Once in every two seconds, a gentle twist of about 10°C should be given to the cylinder. When 50 drops are completed, raise the cylinder to eye level and read the volume of the material.

A-9.3 Calculation

$$\text{Bulk density, g/ml} = \frac{40}{V}$$

where

V = volume in ml of the material.

A-10. FLOW POINT TEST

A-10.0 Outline of the Method — Measured increments of water are added to the material in a test-tube and the quantity of water required to make the paste flow determined.

A-10.1 Procedure — From a 50-ml burette, introduce a volume of water equal to about four-fifths of the expected flow point into a thick walled test-tube (internal dimensions 20 × 150 mm), fitted with rubber bung. Weigh 15·00 g of the material on to a sheet of black glazed paper and add a little at a time to the test-tube. After each addition, replace the bung and shake vigorously. When the paste gets stiff and does not rattle on shaking, add further quantities of water in one millilitre portions alternately with a portion of the remaining material until all of the material is in the test-tube and has formed a paste of stiff consistency. Now add the water in 0·5 ml portions with vigorous shaking between additions (shaking may be best done by pounding over rubber pad or folded duster). To test when the flow point has been reached, give the test-tube a few gentle taps on the bench to shake the paste to the bottom and then invert at an angle of about 15° from the vertical and rotate slowly (do not give the tube any movement to force or shake out the paste). After five seconds, find out whether the paste flows out evenly to the other end of the tube. Note the total volume of water used when this stage is reached.

NOTE — The flow should not be sluggish and the slurry should be free from lumps. If the test-tube is rotated slowly at the flow point in a horizontal position the slurry should flow down the walls and should not rotate with the test-tube.

A-11. TEST FOR CARBONATES

A-11.1 Reagent

A-11.1.1 *Concentrated Hydrochloric Acid* — *see* IS : 265-1976*.

A-11.2 Procedure — To 1 g of the material, add 5 ml of water and 2 ml of concentrated hydrochloric acid.

A-11.2.1 The material shall be taken to have passed the test if no effervescence occurs.

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*Specification for hydrochloric acid (*second revision*).

A-12. TEST FOR IRON

A-12.0 Outline of the Method — The colour produced with thiocyanate is matched against that obtained with standard iron solution.

A-12.1 Apparatus

A-12.1.1 *Nessler Cylinders* — 50 ml capacity.

A-12.2 Reagents

A-12.2.1 *Concentrated Hydrochloric Acid* — *see* IS : 265-1976*.

A-12.2.2 *Ammonium Persulphate or Potassium Persulphate*

A-12.2.3 *Potassium Thiocyanate Solution* — 20 percent.

A-12.2.4 *iso-Butanol*

A-12.2.5 *Standard Iron Solution* — Dissolve 0·700 g of ferrous ammonium sulphate [$FeSO_4.(NH_4)_2SO_4.7H_2O$] in 10 ml of dilute sulphuric acid (10 percent *v/v*) and dilute with water to 1 000 ml. Take 10 ml of the solution and dilute to 100 ml. One millilitre of this solution contains 0·01 mg of iron (as Fe).

A-12.3 Procedure — Dissolve 5·000 g of the material in 50 ml of water and 25 ml of concentrated hydrochloric acid. Add 0·5 g of ammonium or potassium persulphate, mix, heat to boil for about one minute and then cool. Make up the volume with water to 250 ml. Transfer 50 ml of the solution into a 100 ml separating funnel, add 10 ml of potassium thiocyanate solution and then 15 ml of *iso*-butanol. Stopper the funnel, shake vigorously and allow the layers to separate. Transfer the aqueous layer to a second separating funnel and extract again with 15 ml of *iso*-butanol. Transfer the butanol, layer to the first separating funnel and extract the aqueous layer once again with 15 ml of *iso*-butanol. Combine all the butanol layers in a Nessler cylinder, make up the volume to 50 ml with *iso*-butanol and then mix. Carry out a control test using 5 ml of standard iron solution in place of the material and the same quantities of other reagents. Compare the colour produced in the two tubes after two minutes.

A-12.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the control test.

A-13. TEST FOR CHLORIDES

A-13.0 Outline of the Method — Turbidity produced with silver nitrate is compared with that obtained with standard hydrochloric acid.

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*Specification for hydrochloric acid (*second revision*).

A-13.1 Apparatus

A-13.1.1 *Nessler Cylinders* — 50 ml capacity.

A-13.2 Reagents

A-13.2.1 *Dilute Nitric Acid* — approximately 4 N.

A-13.2.2 *Silver Nitrate Solution* — 2 percent.

A-13.2.3 *Standard Hydrochloric Acid* — exactly 0·02 N.

A-13.3 Procedure — Weigh 0·300 g of the material and add to it 10 ml of water and 2 ml of dilute nitric acid. Warm gently, if necessary, to dissolve the material. Filter if necessary, transfer to a Nessler cylinder, dilute to 50 ml and add 1 ml of silver nitrate solution. Mix the contents of the cylinder well and allow to stand for 5 minutes protected from direct sunlight. Carry out a control test in another Nessler cylinder using one millilitre of standard hydrochloric acid in place of the material and compare the turbidity in the two cylinders.

A-13.3.1 The material shall be taken to have passed the test if the turbidity with the material is not greater than that produced in the control test.

A-14. TEST FOR SULPHATES

A-14.0 Outline of the Method — Turbidity produced with barium chloride is compared with that obtained with standard sulphuric acid.

A-14.1 Apparatus

A-14.1.1 *Nessler Cylinders* — 50 ml capacity.

A-14.2 Reagents

A-14.2.1 *Dilute Hydrochloric Acid* — approximately 4 N.

A-14.2.2 *Barium Chloride Solution* — 10 percent.

A-14.2.3 *Standard Sulphuric Acid* — exactly 0·1 N.

A-14.3 Procedure — Weigh 1·000 g of the material and dissolve in the smallest possible amount of dilute hydrochloric acid. Dilute in a volumetric flask of 100 ml. Transfer 20 ml of the solution, previously filtered, if necessary, to a Nessler cylinder, add one millilitre of barium chloride solution and dilute to the mark. Mix the contents of the cylinder and allow to stand for 10 minutes. Carry out a control test in another Nessler cylinder using one millilitre of standard sulphuric acid in place of the material, and compare the turbidity in the two cylinders.

A-14.3.1 The material shall be taken to have passed the test if the turbidity with the material is not greater than that produced in the control test.

A-15. TEST FOR BARIUM

A-15.1 Reagents

A-15.1.1 *Dilute Hydrochloric Acid* — approximately 4 N.

A-15.1.2 *Potassium Sulphate Solution* — 1 percent.

A-15.2 Procedure — Weigh 0·50 g of the material, add to it 10 ml of water and then dilute hydrochloric acid dropwise, stirring after each addition until the material is dissolved. Filter if necessary and add to the filtrate 2 ml of potassium sulphate solution. Allow to stand for 10 minutes.

A-15.2.1 The material shall be taken to have passed the test if no turbidity is produced.

A-16. TEST FOR FLUORIDES

A-16.0 Outline of the Method — The colour (red to yellow with increasing concentrations of fluoride) obtained with thorium alizarin reagent is matched titrimetrically against standard fluoride solution.

A-16.1 Apparatus

A-16.1.1 *Nessler Cylinders* — 50 ml capacity.

A-16.2 Reagents

A-16.2.1 *Perchloric Acid* — 70 to 75 percent (*m/v*).

A-16.2.2 *Sodium Alizarinsulphonate Solution* — Dissolve 0·1 g of sodium alizarinsulphonate in 100 ml of water and filter if necessary.

A-16.2.3 *Standard Sodium Hydroxide Solution* — 0·05 N.

A-16.2.4 *Standard Hydrochloric Acid* — 0·1 N.

A-16.2.5 *Thorium Nitrate Solution* — Dissolve 0·25 g of thorium nitrate in 1 000 ml of water.

A-16.2.6 *Standard Fluoride Solution* — Dry about 0·5 g of sodium fluoride at 200°C for 4 hours. Weigh 0·442 g of the dried sodium fluoride and dissolve it in sufficient water to make 100 ml. Dilute exactly 10 ml of the solution with water to make 1 000 ml. Each millilitre of this solution corresponds to 0·02 mg of fluoride (as F).

A-16.3 Procedure — Place 2·000 g of the material, 5 ml of perchloric acid, 15 ml of water and a few glass beads in a 50-ml distilling flask connected with a condenser and carrying a thermometer and a capillary tube, both of which shall extend into the liquid. Connect a small dropping funnel filled with water to the capillary tube. Support the flask on an asbestos sheet with a hole which exposes about one-third of the flask to the flame. Distil until the temperature reaches 135°C, receiving the distillate under the surface of a few millilitres of water, then maintain a temperature between 135° to 140°C by adding water from the funnel. Continue the distillation until 70 ml have been collected. Dilute the distillate to 80 ml and mix well. Place 40 ml of the solution in one of the Nessler cylinders. In the other Nessler cylinder, place 40 ml of water as a control. Add to each Nessler cylinder 0·1 ml of sodium alizarinsulphonate solution and mix well. Add, dropwise and with stirring, standard sodium hydroxide solution to the Nessler cylinder containing the distillate until its colour just matches that of the control, which is faintly pink. Then add to each Nessler cylinder exactly 1 ml of standard hydrochloric acid and mix well. From a burette, graduated in 0·05 ml, add slowly to the Nessler cylinder containing the distillate enough thorium nitrate solution so that, after mixing, the colour of the liquid just changes to a faint pink. Note the volume of thorium nitrate solution added, add exactly the same volume to the control, and mix. Now add to the control cylinder standard fluoride solution from a burette to make the colours of the two Nessler cylinders match after dilution to the same volume. Mix well, and allow all air bubbles to escape before making the final colour comparison. Check the end point by adding one or two drops of standard fluoride solution to the control; a distinct change in colour should take place.

A-16.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if not more than 5 ml of standard fluoride solution are required for the control test.

APPENDIX B

(*Clause* 4.1)

SAMPLING OF DICALCIUM PHOSPHATE FOR DENTIFRICE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall be taken in a protected place not exposed to damp air dust or soot.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in clean, dry, air-tight glass or other suitable containers on which the material has no action.

B-1.6 Each sample container shall be sealed air-tight with a stopper after filling, and marked with full details of sampling, the date of sampling, and the year of manufacture of the material.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 Samples shall be tested for each lot for ascertaining the conformity of the material to the requirements of this specification.

B-2.2 The number (*n*) of containers to be chosen from the lot shall depend on the size of the lot (N) and shall be in accordance with col 1 and 2 of Table 2.

B-2.3 The containers to be selected for sampling shall be chosen at random. In order to ensure the randomness of selection, random number tables shall be used. For guidance and use of random number tables, IS : 4905-1968* may be referred. In the absence of random number tables the following procedure may be adopted:

> Starting from any container in the lot count them as 1, 2, 3,.... up to *r* and so on in one order, where *r* is the integral part of N/n. N being the lot size and *n* the sample size. Every *r*th container thus counted shall be withdrawn from the lot so as to constitute the required sample size.

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*Methods for random sampling.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED

(*Clause* B-2.2)

Lot Size N	No. of Containers to be Selected n
(1)	(2)
Up to 15	2
16 „ 25	3
26 „ 50	4
51 „ 100	7
101 and above	10

B-3. TEST SAMPLES AND REFEREE SAMPLES

B-3.1 Preparation of Test Samples

B-3.1.1 Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected (*see* Table 2). The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics given under **2** and shall not exceed 0·5 kg.

B-3.1.2 Thoroughly mix all portions of the material drawn from the same containers. Out of these portions, a small but equal quantity shall be taken from each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 600 g. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.2 Referee Sample — The referee sample shall be marked for this purpose and shall bear the seals of the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of a dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Tests for the determination of all the characteristics given in **2** shall be conducted on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 A lot shall be declared as conforming to this specification if the test results on the composite sample meet the corresponding requirements specified in **2**.

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